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Chapter #

RECOVERY OF VOC FROM OFFSHORE AND ONSHORE SHUTTLE TANKERS USING STRUCTURED Y-TYPE ZEOLITE MEMBRANES.

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Abstract: The emissions of volatile organic compounds (VOCs) from onshore and offshore facilities are studied and an alternative technology for the recovery of methane and propane by the use of membrane technology is explored. Permeation tests were carried out with a zeolite membrane consisting of an α – Al_2O_3 support. The permeance of nitrogen, carbon dioxide, helium, methane and propane through the membrane at varying pressures was determined. The permeance of CH_4 was in the range of 1.44×10^{-6} to 3.41×10^{-6} $mol s^{-1} m^{-2} Pa^{-1}$ and a CH_4/C_3H_8 selectivity of 3.3 at 293 K was obtained. The molar flux of the gases was found to have an average linear regression coefficient value R^2 of 0.9892. On the basis of the results obtained it can be concluded that separation of the hydrocarbon gases can be achieved with the zeolite membrane. The main mechanism governing the flow of gases through the zeolite membrane was molecular sieving although there is evidence of deviation from this mechanism. To achieve higher selectivity of the target gas there is need for further modification of the membrane. The morphology of the membrane was determined using the scanning electron microscope, which showed the pore size of the membrane and the support layer.

Keywords: Characterization, Gas separation, Membranes, Shuttle tankers, Volatile Organic Compounds and Zeolite.

1. INTRODUCTION

Volatile organic compounds are chemicals that have high vapor pressure at room temperature. Due to their high vapor pressures, large numbers of molecules evaporate from the initial form of the compound and enter into the atmosphere.

The chemicals in the Volatile organic compounds react in the presence of nitrogen oxides and sunlight to form smog (ground-level ozone), which affect the respiratory system of humans. VOCs are organic chemicals containing the element carbon in their molecular structure and are odorless, colorless and tasteless. Volatile organic Compounds (VOCs) are released during the storage, loading and offloading of hydrocarbon products in onshore and offshore facilities. Methane, ethane and propane are light hydrocarbons and, are considered to be the volatile organic compounds. These compounds are harmful to the environment; methane is a potent greenhouse gas, which contributes to ozone layer depletion. Other Non-methane VOCs (NMVOCS) such as ethane, butane, hexane, pentane and propane react with nitrous oxide to form ground level ozone, which affects both human and plants [1].

Apart from the VOC's being harmful to the environment, they constitute significant economic value that should not be wasted. The sources of the release of VOCs in the onshore and offshore facilities to the atmosphere mostly occur during production, transportation and storage of crude oil [1]. The light hydrocarbons mostly vaporize out of the crude oil during the loading and unloading operations of shuttle tankers and also from Floating Production Storage and Offloading units (FPSOs).

Due to the adverse effects of Volatile Organic Compounds (VOCs) in the environment and also the economic loss of these hydrocarbon compounds, some technologies are used for the abatement of VOCs that are released from both onshore and offshore facilities. Some of the commercially viable methods for treating VOCs emissions include; absorption, adsorption, thermal oxidation, membrane separation and cryogenic condensation.

VOCs Emission Sources

VOCs are emitted to the atmosphere during, production, storage and transportation of crude oil. The two main sources of light hydrocarbon emissions in oil and gas production can be found in onshore and offshore facilities.

Factors affecting the Rate of VOC Emission from onshore and offshore facilities

The rate of emission of Volatile Organic Compounds depends on various factors. These factors are discussed below:

Loading Time

The emission of hydrocarbon gas from a cargo tank is a non-equilibrium process and hence the emission will increase with increase in loading time. The longer the loading operation, the higher the percentage of HC gas emitted [2].

Effects of Ship movement

During loading operations, the movement of the ship can alter the emission rates, also movements due to weather tends to increase circulation in the cargo tank between the crude oil and the atmosphere thus increasing a higher blending between HC vapor and IG which in turn increases the vaporization rate of the HC. This is more prevalent when the shuttle tanker is short loaded and the rolls or pitch could cause more splashing [2].

Nature of Hydrocarbon

A high concentration of light hydrocarbon affects the rate of VOC emitted, since the vaporization rate of the hydrocarbon is increased which thus lead to more hydrocarbon being emitted to the atmosphere. Crude oil composition with high concentration of light hydrocarbons such as methane and ethane tend to have high hydrocarbon vapor, which can mix with inert gas as a result of convection, and then cause high rate of hydrocarbon vaporization from the loading facilities [3].

Temperature of Crude Oil

The temperature of crude is a very important factor in oil and gas production. The Reid Vapor Pressure (RVP) of crude oil is determined at a specific temperature (37.8˚C), which makes RVP independent of temperature. The difference in temperature of the ship and the shore tank leads to a variation between the volumes of liquid loaded and vapor displaced. The vapor displaced into the shore tank expands and warm up when the ship's tank is colder than the shore tank [4]. This leads to subsequent emission and increase in pressure inside the tank.

VOC Reduction Techniques

There are various available techniques for treating VOC emissions from onshore and offshore facilities during the loading and unloading operations. Some of these technique include; absorption, thermal oxidation, adsorption, condensation, reducing volatility and membrane separation [4].

Absorption

Absorption method for VOC recovery is a technology developed by cool sorption. It is basically used to recover non-methane VOCs in chilled liquid or pressurized crude oil (8-11 bar) [5]. This process involves feeding the bottom of the packed column with vapor from the tank during loading operation. The vapor moves upward and is in counter current contact with the chilled liquid absorbent flowing downwards. The absorbent dissolves the hydrocarbon from the vapor/air liquid and removes it from the mixture. The residual air is vented to the atmosphere as it moves out of the top of the column. There is regeneration of the absorbent liquid in the stripping section of the absorption system [4]. Methanol is injected into the absorption system to prevent the formation of hydrates in the vent gas.

Thermal Oxidation

Thermal oxidation method of controlling VOC emission is also called combustion method and is widely used in USA. The combustion system ranges from simple enclosed fares to catalytic oxidizers with internal heat recovery. The issue of safety and combustion emission of CO_2 are the major negative attributes of thermal oxidation. Energy recovery is used to minimize the implication of CO_2 emission while the use of enrichment dilution, detonation arrestors coupled with effective management procedures ensures safety of the system [4].

Adsorption

Adsorption system is mainly used to separate inert gas from hydrocarbon fractions. There are various adsorption systems, one of which is the use of activated carbon. In this type of adsorption technique, the carbon absorbs the organic molecules and the gases like CO_2 and air move through the bed unabsorbed and are emitted to the atmosphere [4]. Before the bed becomes saturated, regeneration is carried out either by vacuum steam stripping, but once the bed becomes saturated, the adsorption process stops and the vapor moves through the bed directly without being absorbed. The use of two beds increases the efficiency of the adsorption process. This continuous operation process involves the use of one bed for the operation, while the other bed is being regenerated.

Cryogenic Condensation

This is method of VOC reduction involving the passing a mixture of VOC containing gas through a liquid nitrogen cooled condenser [4]. This technology is often used in pharmaceutical industry. It is a cost-effective technique of VOCs emission control when compared with the other technologies already discussed [6]. A mathematical description of the process can be developed in order to design a counter current single tube condenser using nitrogen vapor as coolant [7].

Reducing Volatility

This is one of the simplest ways to reduce VOC emissions. It entails the reduction of the volatility of the cargo. Although the vapor pressure of pure substances cannot be altered, changing the composition of the crude to include more of heavier molecular weight compounds and less of lighter molecular weight ones can reduce the gasoline vapor pressure. The volatility of the crude should be reduced before loading and storage.

Sequential Transfer

In this system, additional pipelines are installed on the shuttle tank and are used during loading and offloading of the tank. The shuttle tank is divided into sections and the loading and discharging is done sequentially. The gas outflow from the first loaded section is piped to the bottom of the next section to be loaded, this is repeated sequentially for the remaining sections and the cargo tank is connected to a VOC recovery plant, thus the IG content emitted is considerably low in this system which improves the operating conditions of the recovery plant [2].

Membrane Separation

Membrane technology can be used for the separation of hydrocarbons from inert gases and the different hydrocarbon gases emitted can be individually separated. The separation concept of hydrocarbons from a shuttle tanker using membrane technology requires another process for the recovery of the VOC such as condensation or adsorption [2]. Modern membrane technology can also be used in implementing Process intensification (PI) which is an innovative design method that is aimed at decreasing production cost, waste generated and size of equipment used as well as energy utilization [8]. This process is a pressure driven process that has numerous industrial applications in chemical and petro chemical industries including petroleum refineries [9]. In recent years there has been an increase in demand for light hydrocarbons for use as petrochemical feed stocks and for fuel. This has been met to a large extent by the recovery of these components from petroleum gases and natural gas by the use of a depropanizer [10]. The use of membranes for gas separations is growing at a slow but steady rate [11]. Baker in 2002 made an estimate that the market scale of gas separations using membrane technology by the year 2020 will be five times that of year 2000 [11]. Microporous inorganic membranes having pore sizes of less than 1 nm have been studied extensively for gas separation applications because of their good resistance to harsh chemicals, good thermal and mechanical stability as well as stability under high pressure when compared to polymeric membranes [12]. Today much of the research work is being directed towards the investigation of new membrane material and the development of new membrane structures that exhibit both higher selectivity and permeability of the target gases [13].

Different types of membranes have been studied for the separation of VOCs from inert gases. This process involves the dissolving of vapor molecules in the membrane, which moves by the principle of diffusion to the other end and desorb into the membrane support material [14]. The principle of pressure differential drives the diffusion process. For mesoporous membranes, separation is based on the collision between the gas molecule and the membrane pore wall and hence the mean free path of the gas molecules is greater than the pore size. The diffusion here is governed by Knudsen mechanism and the rate of transport of any gas is inversely proportional to the square root of its molecular weight [14]. However, for a micro porous membrane with pore size less than 2 nm, separation of gases is based mostly on molecular sieving. The transport mechanism in these membranes is often complex and involves surface diffusion that occurs when the permeating species exhibit a strong affinity for the membrane surface and thus adsorbed on the walls of the pores [14].

A membrane's permeance and selectivity has an influence on the economics of a gas separation process [13]. Permeance is the rate at which a substance permeates through a membrane and is dependent on several factors like the pore size and material of the membrane. The selectivity of a membrane is the fundamental parameter to achieving high product purity and high recoveries; hence for the potential growth of membrane gas separation process, the production of highly selective membranes for the desired gas is essential. The development of inorganic membranes like silica and zeolite has increased the potential of membrane gas separation applications as they can withstand aggressive chemicals as well as high temperatures. However, there are drawbacks on the use of such membranes, which includes their high cost, modest reproducibility

The permeance P ($molm^{-2}s^{-1}Pa^{-1}$) represents the proportionality coefficient with the flux at steady state of a particular gas through a membrane and is given by:

$$P = \frac{Q}{A.\Delta p}$$

Where Q is the molar gas flow rate through the membrane ($mol\ s^{-1}$), A is the membrane surface area (m^2) and Δp is the pressure difference across the membrane (Pa). The permeance is therefore a measure of the quantity of a component that permeates through the membrane [15].

The calculated gas selectivity is the ratio of the permeability coefficients of two different gases as they permeate independently through the membrane is given by:

$$\alpha_{ij} = \frac{P_i}{P_j}$$

Where P_i and P_j is the permeance of the single gases through the membrane.

The selectivity is the measure of the ability of a membrane to separate two gases and it is used to determine the purity of the permeate gas as well as determine the quantity of product that is lost.

Gas separation can be used for various applications such as pollution control, photochemical process, oxygen enrichment, pharmaceutical process and many more [16].

This research adapts the use of Y-type Zeolite membrane for the separation and subsequent recovery of hydrocarbon gases under varying conditions of temperature and pressure since zeolites have the ability to withstand high temperature and pressure as well as fine pore size distribution that is highly selective to hydrocarbon gases.

2. EXPERIMENTAL

Permeation setup

A schematic diagram of the membrane flow apparatus used for the permeation test for the gases is presented in Fig. 1. Four different gases: carbon dioxide, helium, nitrogen, methane and propane were used for the permeability through a porous zeolite membrane at various transmembrane pressures.

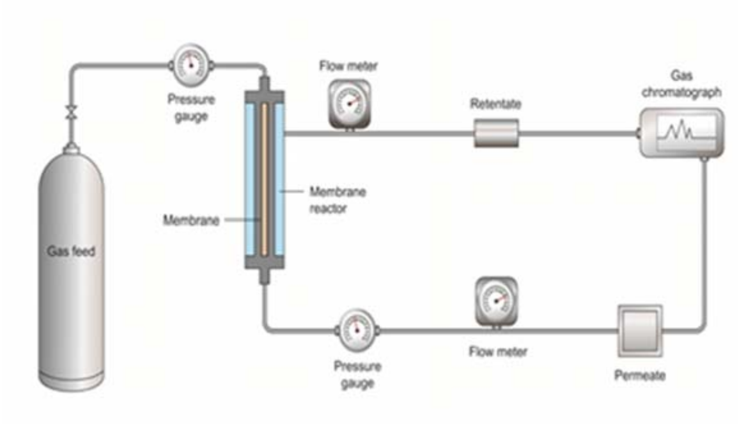

Fig 1: Schematic diagram of a membrane permeation flow system

Membrane Preparation

A solution containing Silicone oxide, aluminum oxide, sodium oxide and deionized water was prepared and homogenized at room temperature for 20 hours; the amount of each substance used is given in Table 1. Zeolite crystals were deposited on alumina support, which is subsequently dipped into the solution and kept for 20 hours at 343 K. The membrane was washed with deionized water and the pH of the rinse water was monitored. When the rinse water pH was neutral the membrane was air dried for 20 minutes and thermally treated in the oven at 338 K for 2 hours prior to permeation test [17].

Table 1: Composition of the modification solution for zeolite membrane

Chemical	Amount (ml)
Aluminium oxide	10
Sodium hydroxide	14
Deionised water	798
Silicone oxide	1

Membrane Characterization

The morphology of the membrane was determined by the use of the Zeiss EVO LS10 scanning electron Microscope. Nitrogen physisorption measurements were carried out at 77.35 K using a Quanta chrome adsorption gas analyzer. The operating conditions of the instrument in given in table 2.

Table 2: Optimum operating conditions of the Quanta Chrome Gas Analyzer

Parameter	Value
Area (A^2mol$_{-1}$)	16.2
Non-Ideality	6.58 x 10 -5

(1/mmHg)	
Sample cell type (mm)	12
Analysis time (mins)	237
Mol weight ($gmol^{-1}$)	28.0134
Ambient temperature (K)	300
Bath temperature (K)	77

The experimental rig (fig. 1) was used to determine the permeance of the gases. The gases were fed to the zeolite membrane from the gas cylinder through the gas inlet; the pressure was controlled at the inlet port by a pressure gauge. The permeate flow rate of the individual gases was measured by a digital flow meter in liters per minute.

3. RESULTS AND DISCUSSION

The zeolite membrane showed permeance in the range of 10^{-6} $molm^{-2}s^{-1}Pa^{-1}$ for CO_2, CH_4, He and N_2, but in the range of 10^{-7} for propane. These permeances are relatively high when compared to literature values [18]. The maximum selectivity for this membrane was calculated and presented in Table 3.

Table 3: Maximum selectivity of methane through a zeolite membrane at 293 K

Gas mixture	CH_4/CO_2	CH_4/C_3H_8	CH_4/N_2	CH_4/He
Selectivity	2.9	3.3	1.4	1.2

The selectivity of methane over propane (Table 3) is higher than the values ranging from 1.42 to 2.56 obtained from the work of Tirouni, Sadeghi and Pakizeh (2015) [19].

The effect of the kinetic diameter on the permeance of the gases is depicted in Fig. 2. The order of the kinetic of the gases is: $He<CO_2<N_2<CH_4<C_3H_8$. Hence, the separation of helium, nitrogen and propane as observed in Fig. 2 was based on molecular sieving properties of zeolite. Carbon dioxide and methane deviated from the expected pattern. This could indicate the presence of inter-crystalline defects in the zeolite membrane [20].

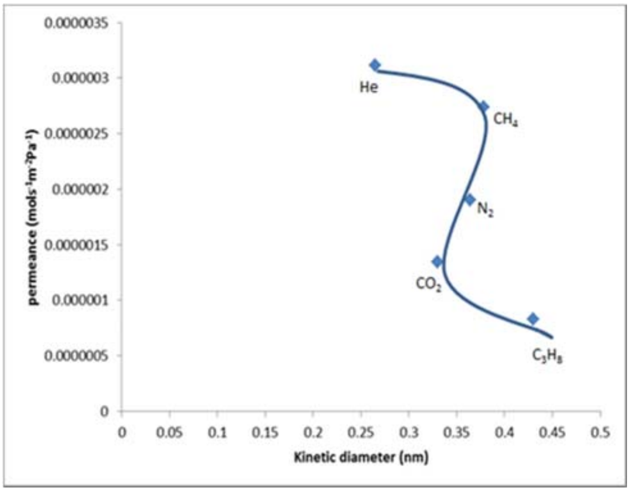

Fig. 2: Effect of kinetic diameter on gas permeance at 293 K and 10^4 Pa

The molar fluxes are linear functions of the pressure drop across the zeolite membrane [19]. Figure 3 shows the molar fluxes of the gases increase linearly with the increase in pressure. Pressure has significant effect on the gas flux (Fig. 3). The difference between the fluxes of the gases increases with increase in pressure, rate of increase of the flux of carbon dioxide, propane, methane, oxygen and nitrogen was observed to have slightly reduced at a higher of pressure of about 1.0×10^5 Pa and higher. The contribution of viscous flux to the overall mass transfer at higher pressure for zeolite membrane might have caused this. A good linear regression value in the range of $R^2 = 0.99$ was observed.

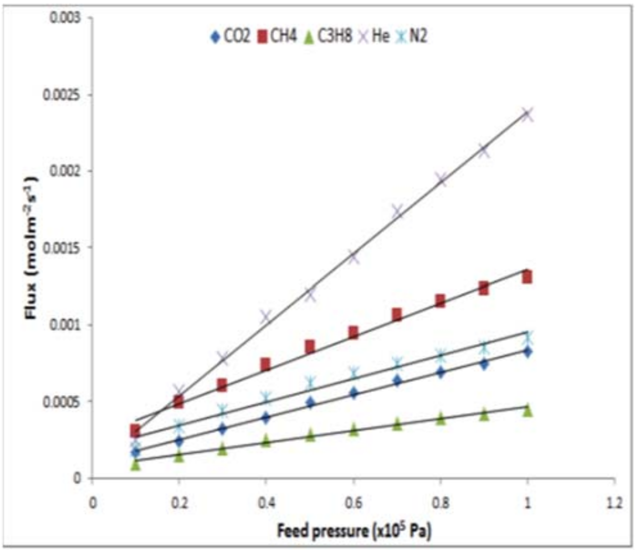

Fig. 3: Molar flux of the gases through zeolite membrane at 293K

Figure 4 (a) and (b) shows the cross sectional view and outer surface of the zeolite membrane. The surface of the membrane is covered with a dense layer of zeolite crystals.

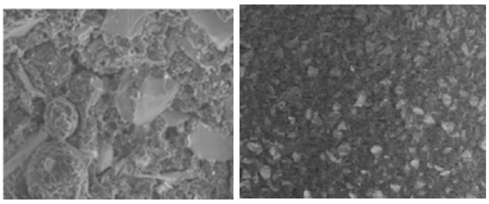
(a) (b)

Fig. 4: SEM images of the cross sectional view (a) and outer surface (b) of zeolite membrane

Fig. 4 clearly shows the zeolite crystals that are deposited on the alumina support. The cross sectional and outer images show a good bonding of the zeolite to the support. The cross sectional view showed the formation of an intermediate layer on the support. This might have led to an improvement in the affinity amidst the zeolite top layer and the alumina support. The zeolite crystals deposited on the surface of the alumina support is visible from the micrograph in the outer surface image in figure 4(b). The pore sizes seen on the outer image was observed to be larger, this could have been caused by the result of interaction of the solution used to coat the membrane surface. It can be observed that the pore diameter is not uniformly distributed.

The hysteresis isotherm in Figure 5 implies the membrane is mesoporous and could undergo capillary condensation during hysteresis. Table 4 shows the desorption summary of the membrane.

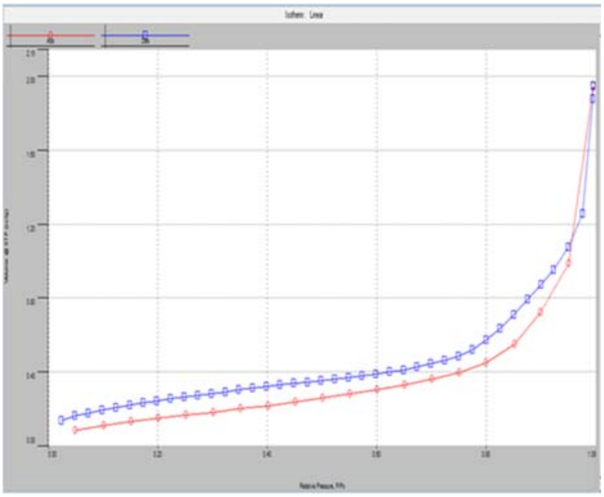

Fig. 5: Physisorption isotherm of the zeolite membrane

Table 4: Desorption summary of the zeolite membrane

Parameter	Value
Pore Volume (cc/g)	0.003
Pore Diameter Dv(d) (nm)	3.94
Surface Area (m^2g^{-1})	0.520

4. CONCLUSION

The emission of volatile organic compounds from shuttle tankers does not only cause harmful effects to the environment but also causes huge monetary loss. The use of membrane technology is one of the emerging technologies that can be used for the recovery of volatile organic compounds. This recovery is based on the permeances and separation factor of the gases through the membrane. Previous studies have shown that membrane material used for gas separation affects the separation factor of that membrane. Zeolites in this work have proved to be a good choice of membrane material for the possible use on offshore and offshore storage facilities. Further work needs to be done for the synthesis of a defect-free membrane that is reproducible and can be introduced into the petroleum and gas industries for the separation of lower hydrocarbons at a competitive cost.

NOMENCLATURE

FPSOs	Floating Production, Storage Offloading units
HC	Hydrocarbon
IG	Inert gas
NMVOC	Non-methane volatile organic compound
PI	Process Intensification
RVP	Reid Vapour Pressure
VOC	Volatile organic compound

Symbols

A = Area of the membrane (m^2)
P = Permeability (mol m m^{-2} s^{-1} Pa^{-1})
Q = Molar gas flow rate (mol s^{-1})
R = Molar gas constant (8.314 J mol^{-1} K^{-1})
ΔP = Pressure drop across the membrane (Pa)
$\alpha_{i,j}$ = Selectivity of component *i* over *j*

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